



TRANSMITTAL LETTER

In Application of:
ANS WESTMIJZE et al.

Serial No: 09/889,436

Filing Date: October 13, 2001

Title: AQUEOUS PEROXIDE EMULSIONS

Assistant Commissioner for Patents
Washington, D.C. 20231

Docket No: ACD 2665 US

Examiner: M. L. Reddick

Group Art Unit: 1713

CERTIFICATE OF MAILING

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☒ Applicant hereby petitions for an extension of time under 37 CFR 1.136 of:

☒ One Month (\$110.00)

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Respectfully submitted,

Richard P. Fennelly
Richard P. Fennelly
Attorney for Applicant(s)
Reg. No. 25,677

03/28/2003 AWONDAF1 00000052 011350 09889436
02 FC:1251 110.00 CH

Akzo Nobel Inc.
Intellectual Property Department
7 Livingstone Avenue
Dobbs Ferry, NY 10522-3408
Tel No.: (914) 674-5464



Patent

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IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

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APPEAL BRIEF

This is an appeal from the Final Rejection of Claims 16-29, dated November 6, 2002.

The text of the rejected Claims is reproduced in the Appendix attached as last section to this Appeal Brief.

REAL PARTY IN INTEREST

The real party in interest for this Appeal is Akzo Nobel N.V., Arnhem, The Netherlands.

RELATED APPEALS AND INTERFERENCES

There are no related appeals or interferences to the instant appeal.

STATUS OF CLAIMS

Claims 16-29 are pending in the instant application and all have been rejected.

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STATUS OF AMENDMENTS

All Amendments have been entered, including an Amendment after Final Rejection wherein new Claims 16-29 were added to the application in place of original Claims 1-15.

SUMMARY OF INVENTION

The present invention relates to an aqueous peroxide emulsion, comprising a peroxide and optionally containing anti-freeze and/or further additives. The peroxide emulsion contains a specific emulsifier system which consists essentially of a copolymer of an α, β -unsaturated dicarboxylic acid and a C_{8-24} α -olefin, the acid groups of which are esterified with an ethoxylated alcohol having a degree of ethoxylation of 1-45. The emulsifier system further comprises an ethoxylated fatty alcohol with an HLB-value greater than 16.

ISSUES

The following two issues are presented for this Appeal:

Whether the Examiner was correct in rejecting Claims 16-29 as obvious over EP 492,712 in combination with PCT WO 98/18835, US 4,499,250, US 4,547,481 or US 4,734,135; and

Whether the Examiner was correct in rejecting in rejecting new Claim 24 under the second paragraph of Section 112.

GROUPING OF CLAIMS

For purposes of the present Appeal, all pending Claims stand or fall together.

ARGUMENT

The Rejection under Section 103

It is well established that to establish a *prima facie* case of obviousness, the Patent and Trademark Office must satisfy *all* of the following requirements. First, the prior art relied upon, coupled with the knowledge generally available in the art at the time of the invention, must contain some suggestion or incentive that would have motivated the person of ordinary skill in the art to modify a reference or to combine references. *In re Fine*, 5 U.S.P.Q.2d 1596, 1598 (Fed. Cir. 1988). Second, the proposed modification must have had a reasonable expectation of success, as determined from the vantage point of one of ordinary skill in the art at the time the invention was made. *Amgen v. Chugai Pharmaceutical Co.*, 18 U.S.P.Q.2d 1016, 1023 (Fed. Cir. 1991, cert. denied, 502 U.S. 856 (1991)). Third, the prior art references or combination of references must teach or suggest all of the limitations of the claims. *In re Wilson*, 165 U.S.P.Q. 494, 496 (C.C.P.A. 1070). In the present situation, the Examiner has not established a *prima facie* case of obviousness.

Cited EP 492,12 discloses a peroxide emulsion comprising a copolymer of an unsaturated dicarboxylic acid and a C₈-C₂₄ olefin, and a second copolymer of at least one polyalkylene and a polysiloxane. The presence of ethoxylated fatty alcohols in the emulsifier system is neither mentioned nor suggested by this reference.

PCT WO 98/18835 describes a peroxide emulsion comprising one or more polyvinyl alcohols and one or more emulsifiers which may be an ethoxylated fatty alcohol. Hence, if the teachings of EP 192,712 and PCT WO 98/18835 were to be combined, a peroxide emulsion would be obtained that contained at least a copolymer of an unsaturated

dicarboxylic acid and a C₈-C₂₄ olefin, a copolymer of at least one polyalkylene and a polysiloxane, a polyvinyl alcohol and an ethoxylated fatty alcohol.

In order to arrive at the subject matter of Claim 16, the person of ordinary skill in the art has to specifically choose to leave out from the composition the copolymer of polyalkylene and polysiloxane and polyvinylalcohol, although neither of the above-mentioned documents gives any direct reference or suggestion to help such a person with this choice. Moreover, PCT WO 98/18835 specifically refers to the composition described in EP 492,712 as being an unacceptable formulation of polyvinyl chloride, thus indicating that the person of ordinary skill in the art, when looking at the EP document, is looking in the wrong direction. Consequently, such a person would never combine these two documents, as the Examiner has done.

The person of ordinary skill in the art would also not derive the subject matter of new Claim 16 from a combination of EP 492,712 and US 4,547,481 (or US 4,499,250, a divisional thereof). US 4,547,481 describes a peroxide emulsion comprising an ethoxylated fatty alcohol or an ethoxylated fatty acid. If one were to combine the EP document with the teachings of either of these US patents, one first has to choose the ethoxylated fatty alcohol, instead of the ethoxylated fatty acid, and subsequently one has to leave out the copolymer of polyalkylene and polysiloxane in order to arrive at the subject matter of new Claim 16. Since these cited documents do not refer to one another in any way, and since they do teach or indicate to combine both teachings, these choices would not have been to the person of ordinary skill in the art. In addition, US 4,499,250 teaches such a person that ethoxylated fatty acids are preferred over ethoxylated fatty alcohols (see Col. 3, lines 38-40),

thereby making the choice for an emulsion with ethoxylated fatty alcohols even less obvious.

Moreover, a combination of the cited EP document, PCT WO 98/18835 or US 4,547,481/US 4,734,135 does not teach the person of ordinary skill in the art the peroxide emulsion of new Claim 16 either for exactly the same reasons enumerated above. More specifically, the person of ordinary skill in the art would not derive, in an allegedly obvious manner, from such a combination of prior art documents a peroxide emulsion which consists essentially of a copolymer of an α,β -unsaturated dicarboxylic acid and a C_8 - C_{24} α -olefin, the acid groups of which are esterified with an ethoxylated alcohol having a degree of ethoxylation of 1-45.

For the foregoing reasons, the Board is requested to reverse the obviousness rejection that currently exists in regard to Claims 16-29.

The Rejection Under Section 112

The Examiner has argued that the terminology "a viscosity between 10-300 mPa.s" is indefinite "as per the conditions under which such was obtained is not readily ascertainable". The Board is requested to reject this contention and reverse the rejection since it is deemed that the articulation of final viscosity values in this Claim is quite definite especially when that Claim needs to be construed in light of the specification where the conditions are articulated at page 7, lines 2-6. It is not improper form to merely recite the viscosity value as has been done in Claim 24. In this regard the Board is requested to consider the representative and attached Claims section from U.S. Patent Nos. 4,137,213 (see the preamble of Claim 1 where underlined) and 5,286,806 (see Claim 1, subparagraph c) and Claim 2, both where underlined).

For all of the reasons provided herein, the Board is requested to reverse each of the Examiner's rejections.

Respectfully submitted,

Richard P. Fennelly

Richard P. Fennelly
Attorney for Applicant
Reg. No. 25,677

Akzo Nobel Inc.
7 Livingstone Avenue
Dobbs Ferry, New York 10522-3408
(914) 674-5464
2665AB.DOC

APPENDIX

THE REJECTED CLAIMS

-- 16. An aqueous peroxide emulsion, comprising a peroxide and optionally containing anti-freeze and/or further additives, which contains a specific emulsifier system which consists essentially of a copolymer of an α,β -unsaturated dicarboxylic acid and a C_{8-24} α -olefin the acid groups of which are esterified with an ethoxylated alcohol having a degree of ethoxylation of 1-45, characterized in that the emulsifier system further comprises an ethoxylated fatty alcohol with an HLB-value greater than 16. --;

-- 17. The emulsion according to Claim 16 wherein the peroxide is selected from the group consisting of peroxyesters, peroxydicarbonates, peroxycarbonates, diacyl peroxides, and combinations thereof, and in which said peroxide is present in an amount of 30-70% by weight, based on the weight of the emulsion.--;

-- 18. The emulsion according to Claim 17 comprising one or more peroxides which require refrigerated storage and are present in an amount of 40-65% by weight, based on the weight of the emulsion. -
-;

-- 19. The emulsion according to Claim 18 further comprising an anti-freeze selected from the group consisting of methanol, ethanol, isopropanol, ethylene glycol, propylene glycol, and glycerol. --;

-- 20. The emulsion according to any one of the Claims 16-19 wherein the copolymer is present in an amount of 0.05 to 20% by weight and the ethoxylated fatty alcohol is present in an amount of 0.02-15% by weight, with the proviso that the total weight of both

compounds is at least 0.5% by weight, all based on the weight of the peroxide in said emulsion. --;

-- 21. The emulsion according to claim 20 wherein the copolymer is present in an amount of 0.1-15% by weight, based on the weight of the peroxide. --;

-- 22. The emulsion according to any one of the Claims 16-19 wherein the HLB value of the ethoxylated fatty alcohol is greater than 16.5. --;

-- 23. The emulsion according to any one of Claims 16-19 wherein the droplet size of the emulsion, when measured using a Malvern Easy Sizer, is characterized by a d50 of 0.1-2.0 μm and a d99 of 0.5-9.0 μm .--;

-- 24. The emulsion according to any one of Claims 16-19 wherein the ingredients are chosen in such a way that a viscosity between 10-300 mPa.s is obtained. --;

-- 25. A polymerization process comprising polymerizing a monomer in the presence of an emulsion according to any one of Claims 16-19 or a polymerisation modification process comprising treating a polymer with an emulsion according to any one of Claims 16-19. --;

-- 26. Polyvinyl chloride obtained by a process involving the reaction of at least vinyl chloride monomer and peroxide that was used in the form of an emulsion according to any one of Claims 16-19. --;

-- 27. The emulsion according to Claim 17 wherein the peroxide is selected from the group consisting of peroxyesters, peroxydicarbonates, peroxyarbonates, diacyl peroxides, and combinations thereof, requires refrigerated storage and is present in an amount of 50-65% by weight, based on the weight of the emulsion. -
-;

-- 28. The emulsion according to Claim 20 wherein the copolymer is present in an amount of 0.2-10% by weight, based on the weight of the peroxide. --;

-- 29. The emulsion according to any one of Claims 16-19 wherein the HLB value of the ethoxylated fatty alcohol is greater than 17.0. --

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cations DOS Nos. 1,546,840; 1,546,848; 1,814,072, 1,930,949, 2,033,770, 2,057,799, 2,163,143, 2,261,804, 2,236,910, 2,252,536 and 2,363,074 and in particular in German Laid-Open applications DOS No. 2,320,301 (corresponding to U.S. application Ser. No. 461,503), 2,357,075 (corresponding to U.S. application Ser. No. 522,629 or U.S. Pat. No. 3,994,989) and 2,554,080. The binders, in a protonized form, which are described in these publications are reaction products of Mannich bases, produced from polyhydric polynuclear phenols, secondary alkanolamines and formaldehyde, with epoxy resins. They carry ammonium and hydroxyl groups, as well as isocyanate groups which may or may not be blocked.

In the Examples, parts and percentages are by weight.

EXAMPLE

(a) Manufacture of the wetting agent:

1,485 parts of a glycidyl ether or pentaerythritol (epoxide value about 0.57) are reacted with 573.6 parts of phenol in the course of 2 hours at 180° C. The epoxide value drops to about 0.08. The resulting modified epoxy resin B is mixed in various ratios with the tetrabutyl ether of tetramethylolacetylenediurea (component A), 0.1% (based on solids) of p-toluenesulfonic acid is added in each case, and the mixture is heated at 100° C. The results are shown in Table 1.

TABLE 1

Experiment	Ratio A:B	Reaction time hours	Molecular weight	Viscosity mPa.s
I	8:2	2	~800	~600
II	6:4	3	~740	~800
III	4:6	3	~1,1800	~1,000

(b) Manufacture of a pigment paste:

428 parts of wetting agent II, 400 parts of talc, 85 parts of butylglycol and 210 parts of iso-butanol are mixed in a high-speed stirrer and 86 parts of carbon black are then added; the mixture is ground three times on a triple roll mill.

(c) 150 parts of the pigment paste are mixed with 856 parts of the cationic binder described in Example 3 of U.S. Pat. No. 3,994,989 and 12 parts of glacial acetic acid. The mixture is then diluted to 6 liters with fully desalinated water. To remove the solvent, the bath is stirred for 3 days at 30° C.

(d) Electrocoating:

An L-shaped phosphatized piece of sheet iron is electrocoated with the above finish for 2 minutes at 280 volts and 30° C. After curing (190° C./20 minutes), both the vertical and the horizontal faces exhibit a perfect surface.

We claim:

1. A pigment formulation which contains pigments, organic solvents and from 10 to 80% by weight of a wetting agent, wherein the wetting agent is a non-ionic organic compound which has a viscosity of from 500 to 10,000 mPa.s and has been produced by reacting

(A) an alkylated amide/formaldehyde condensation product with

(B) a modified epoxy resin which is an adduct of an epoxy resin and one or more compounds selected from the group consisting of phenol, bisphenol, mercaptan and dimercaptan, at least 80% of the epoxide groups of the epoxy resin having undergone adduct formation, the weight ratio of (A) to (B) being from 9:1 to 1:9 and the reaction of (A) with (B) being carried out at from 80 to 120° C. in the presence of an acid catalyst.

2. A pigment formulation as set forth in claim 1, wherein the component (A) is a condensation product of formaldehyde and a compound from the group comprising melamine, urea, benzoguanamide and acetylenediurea, and has been at least partially etherified with an alkanol of 1 to 8 carbon atoms.

3. A pigment formulation as set forth in claim 1, wherein the wetting agent has been obtained by substitution of the hydroxyl groups of (B) for the alkanols from the ether groups of (A) to a degree of from 10 to 100% by transesterification.

4. A surface-coating binder to which has been added a pigment formulation as set forth in claim 1, the content of wetting agent being from 5 to 20% by weight, based on the binder.

5. A surface-coating binder as set forth in claim 4, said binder being a reaction product of Mannich bases, produced from polyhydric polynuclear phenols, secondary alkanolamines and formaldehyde, with epoxy resins, and said binder being in a protonized form.

6. A cationic electrocoating finish containing a surface-coating binder as set forth in claim 4.

7. A cationic electrocoating finish containing a surface-coating binder as set forth in claim 5.

* * * * *

-continued

Example	30% NaOH (parts)	SV (mPa.s)	Carboxyl Content (mole %)
9	95.7	9.4	9

EXAMPLES 10-12

Example 1 is repeated except that higher amounts of NaOH are used. In addition, part of the samples are heat treated. The result is a substantially greater degree of hydrolysis of the polymers as indicated by the carboxylate values set forth below. In all cases stable emulsions are produced.

Example	NaOH/PAM	SV	CXL(a) (mole %)	CXL(b) (mole %)	CXL(c) (mole %)
10	0.9	9.5	55	63	72
11	1.25	9.1	61	71	84
12	1.5	9.5	64	60	86

CXL = carboxyl (in mole %):

a. reaction at room temp.

b. reaction at 60 degs. C. for 64 hours

c. as b. analysis performed 8 days later

EXAMPLE 13

Example 1 is repeated. The product has a SV of 10.86 and an IV of 50.4 dl/g. [This corresponds to a molecular weight of 60.7 million using formula 1, i.e., $IV = 0.000373 \times \text{molecular weight}^{0.66}$.]

A sample of a commercially available acrylic acid-/AMD copolymer has a SV of 6.08 mPa.s and an IV of 29.7 dl/g (molecular weight=26 million).

EXAMPLE 14

Example 1 is repeated. The polymer has a SV of 10.0. Clay settling tests, as in Example 3, are conducted comparing performance against other anionic PAMS.

Polymer	Dose required to give 10 feet/ hour Settling Rate Dose Relative to Ex. 14
That of Ex. 14	1.0
Competitive dry PAM A.	1.29
Competitive dry PAM B.	1.41
Competitive dry PAM C.	1.397
Competitive emulsion PAM D.	2.05
Competitive emulsion PAM E.	2.68
Competitive emulsion PAM F.	2.71

This example demonstrates that a substantially greater amount of the prior art material is required to attain the same results (i.e., a settling rate of 10 feet/hour) as that achievable with use of the polymer of the present invention.

EXAMPLE 15

Example 1 of Connolly U.S. Pat. No. 4,171,296 is repeated exactly as described. The emulsion product (polymer control Q) has a SV of 6.85 mPa.s (M.W.=about 30 million). A sample of the polymer is precipitated for analysis. The precipitated polymer contains 35 mole % carboxylate.

In addition, Example 1 of the present application is repeated. The SV of the emulsion product so produced is 11.17 mPa.s and the carboxylate content of the precipitated polymer is 35 mole %. Clay settling tests as

described in Example 3 are conducted and the results shown below:

Also, the procedure of Example 1 is followed to produce a lower molecular weight polyacrylamide having a SV of 6.5 mPa.s, i.e., about 28,000,000 M.W., which is then hydrolyzed as described. This PAM is identified below as Polymer B.

Dose lb/ton	Settling Rate (ft/hours)		
	Control Polymer Q	Polymer of Example 1	Polymer B
0.5	—	11.97	8.68
0.75	—	22.77	15.05
1.0	5.48	37.37	24.09
2.00	10.92	—	—
3.00	19.56	—	—

These data clearly demonstrate the superiority of the high molecular weight polymers of the present invention.

EXAMPLES 16 and 17

Example 1 is repeated except that the molecular weight of the polyacrylamide back-bone emulsion reactant is reduced by adding isopropanol chain-transfer agent to the monomer prior to polymerization. The hydrolysis reaction is then carried out as described in Example 1.

Example	Isopropanol (% on monomer)	SV of Final Product	Approx. MW
16	0.75%	6.17	27 million
17	1.75%	4.91	20 million

EXAMPLES 18-23

Example 1 is repeated except that various stabilizing surfactants are added. In addition, an ethoxylated alcohol inverting surfactant is used.

Example	Stabilizer Surfactant	SV of final product
18	isopropylamide of oleic acid*	10.64
19	polyoxyethylene oleyl ether (HLB = 8.2)	10.03
20	polyoxyethylene oleyl ether (HLB = 5.0)	10.74
21	substituted imidazoline of oleic acid	11.11
22	diethanolamide of oleic acid*	10.64
23	polyoxyethylene lauryl ether	10.50

*not alkali stable

While it is apparent that the invention herein disclosed is well calculated to fulfill the objectives stated above, it will be appreciated that numerous modifications and embodiments may be devised by those skilled in the art, and it is intended that the appended claims cover all such modifications and embodiments as fall within the true spirit and scope of the present invention.

We claim:

1. A method of preparing a flocculating agent having improved properties which comprises:

a) providing a water-in-oil emulsion which comprises, as a discontinuous phase, small droplets of an aqueous acrylamide monomer solution containing a redox polymerization catalyst and, as a con-

13

tinuous phase, a liquid hydrocarbon containing an oil soluble emulsifying agent;

b) polymerizing said acrylamide monomer to form aqueous acrylamide polymer droplets as the discontinuous phase; and

c) reacting said acrylamide polymer with an alkali metal hydroxide or quaternary ammonium hydroxide to hydrolyze at least a portion of said polymer to produce a polymer having a molecular weight of at least about 10 million as indicated by an intrinsic viscosity of at least about 15 dl/g. and a solution viscosity of at least about 4 mPa.s.

2. The method of claim 1 wherein said polymer has a molecular weight of at least about 20,000,000 as indicated by an intrinsic viscosity of at least about 32 dl/g. and a solution viscosity of at least about 7 mPa.s.

3. The method of claim 2 which further comprises adding to the emulsion, before step c), a stabilizing

14

compound selected from the group consisting of an oil-soluble emulsifying agent and an organic surfactant.

4. The method of claim 3 wherein said emulsion is stabilized by adding said oil soluble emulsifying agent thereto in an amount of between 0.1 and 30% by weight, based upon the weight of the oil.

5. The method of claim 3 wherein said emulsion is stabilized by adding said organic surfactant thereto in a concentration of from about 0.10 to 15% by weight.

6. The method of claim 1 which further comprises adding an inverting agent to said hydrolyzed high molecular weight PAM dispersion to release said hydrolyzed particles in an aqueous solution.

7. The method of claim 6 wherein said inverting agent is nonylphenyl ethoxylate.

8. The method of claim 1 which further comprises selecting tertiary butyl hydroperoxide-sodium metabisulfite or sulfur dioxide as the redox catalyst.

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